

PATENT ABSTRACTS OF JAPAN

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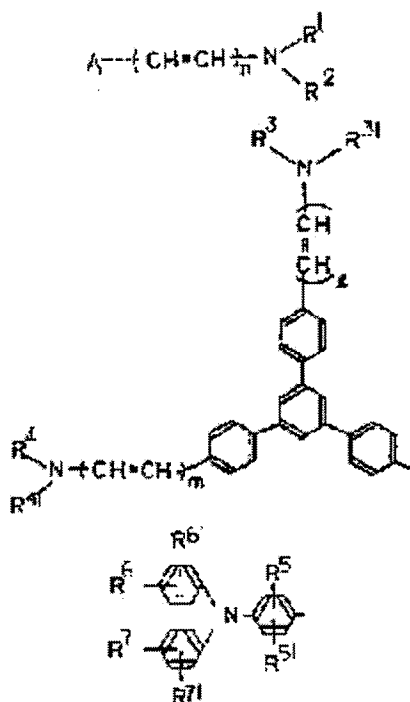
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(54) ORGANIC SOLAR BATTERY



(57)Abstract:

PURPOSE: To obtain a good photoelectric conversion efficiency and stabilize characteristics in a continuous service, by forming a photoconductive layer containing a charge occurrence pigment and a specific charge transport dye between two electrodes. CONSTITUTION: A photoconductive layer containing a charge occurrence pigment and a charge transport dye is installed between two electrodes. The charge transport dye is formed of a compound shown in expression I. In expression I, R^1 and R^2 are an alkyl group or a phenyl group in which a substituent group may be used, and n is an integer of 1 to 10, and A is a group shown in expression II and III. In expression II and III, R^3 , R^4 , R^{31} and R^{41} are the alkyl group or the phenyl group in which the substituent group may be used, and l or m are an integer 0 to 3, and R^5 , to R^7 , R^{51} , R^{61} and R^{71} are an hydrogen atom, the alkyl group or the like. Also, an electric charge occurs in the charge occurrence

pigment, and while an electron is moved to an anode, a hole is injected into the charge transport dye and is moved to a cathode. Thus, characteristics in a continuous service can be stabilized.

LEGAL STATUS

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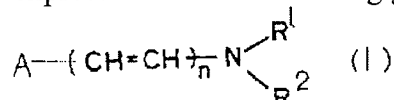
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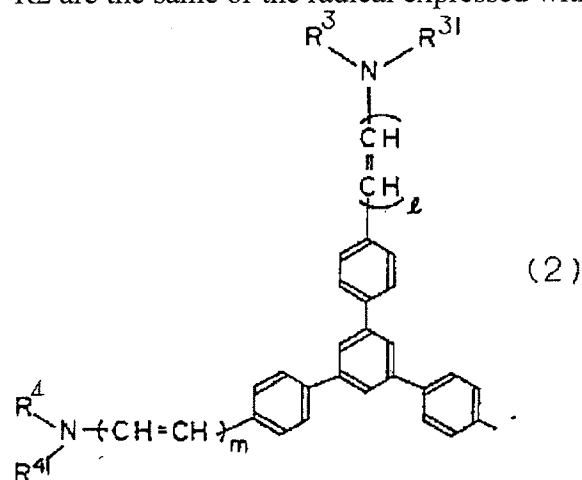
CLAIMS

[Claim(s)]

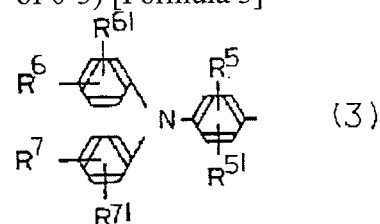
[Claim 1] The organic solar battery this whose charge transportation color it is the organic solar battery with which the photoconductive layer containing a charge generating pigment and a charge transportation color is prepared between two electrodes, and is the compound expressed with the following general formula (1): [Formula 1]



: (A the phenyl group which may differ and may have the alkyl group or the substituent is indicated to be, and n indicates the integer of 1-10 to be shows among a formula that R1 and R2 are the same or the radical expressed with the following type (2) or (3)) [Formula 2]



: (Among a formula, R3, R4, R31, and R41 being the same or the phenyl group which may differ and may have the alkyl group or the substituent is shown, and l and m show the integer of 0-3) [Formula 3]



(the inside of a formula, and R5, R6, R7, R51, R61 and R71 are the same -- or it differs and a

hydrogen atom, an alkyl group, an alkoxy group, an aryl group, or a cycloalkyl radical is shown) .

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the organic solar battery of the functional discrete type using an organic pigment and organic dye in more detail about an organic solar battery.

[0002]

[Description of the Prior Art] An organic solar battery transforms light energy to electrical energy by making pn junction form between the photoconductive layers which consist of an inorganic semi-conductor and organic coloring matter, such as silicon and germanium, or combining a metal and organic coloring matter. The photoconductive layer is what thin-film-ized the organic photo conductor which consists of conductive polymers, such as synthetic colors, such as chlorophyll, and a pigment, polyacethylene, or those composite material by vacuum evaporation technique, the cast method, or the dipping method, and is formed.

[0003] Since the organic solar battery using the above organic photo conductors and organic semiconductors has advantages, such as excelling in economical efficiency, and being easy to manufacture, compared with the inorganic solar battery which made pn junction form in the single crystal of inorganic semi-conductors, such as conventional silicon and germanium, it attracts attention as a noncommercial solar battery in recent years.

[0004] For example, the organic solar battery which arranged in inter-electrode [two] the photoconductive layer which distributed the organic charge generating pigment and the organic charge transportation color in binder resin, and was formed in JP,1-215070,A is indicated.

[0005] In this organic solar battery, organic compounds, such as a pyrazoline system, a hydrazone system, an oxazole system, a thoria reel methane system, and the poly aryl alkanes, are used as an organic charge transportation color.

[0006] Therefore, according to the organic solar battery indicated by this JP,1-215070,A, it is supposed that it is cheap, photoelectric conversion efficiency is high, and an organic solar battery with high continuous duty stability will be obtained.

[0007] In this organic solar battery, the Schottky barrier is formed between the above-mentioned electrode and the photoconductive layer containing the above-mentioned organic charge generating pigment. For this reason, if light is irradiated by the above-mentioned organic charge generating pigment, a carrier pair will occur in this Schottky barrier part. An electron hole is poured into the organic charge transportation color in a photoconductive layer among these carrier pairs, and, as for an electron hole, an electron moves the inside of this color in the inside of the above-mentioned pigment according to the potential difference of the Schottky barrier, respectively.

[0008]

[Problem(s) to be Solved by the Invention] However, carrier mobility is low and the

photoelectric conversion efficiency of the organic solar battery using the above-mentioned organic charge transportation color is inadequate. furthermore, it is easy to deteriorate by UV irradiation, and a property is unstable at the time of continuous duty -- etc. -- it has the trouble.

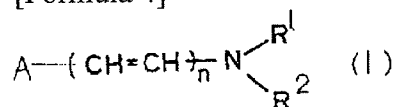
[0009] This invention is to offer the organic solar battery which it is made in order to solve the above-mentioned trouble, and the purpose has a high rate of photo electric conversion, and has a stable property at the time of continuous duty.

[0010]

[Means for Solving the Problem] The organic solar battery of this invention is a hydrazone system compound which is the organic solar battery with which the photoconductive layer containing a charge generating pigment and a charge transportation color is prepared between two electrodes and by which this charge transportation color is expressed with the following general formula (1), and the above-mentioned purpose is attained by that.

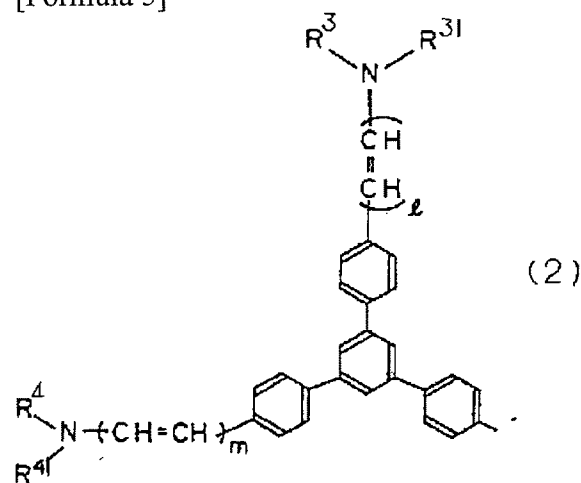
[0011]

[Formula 4]



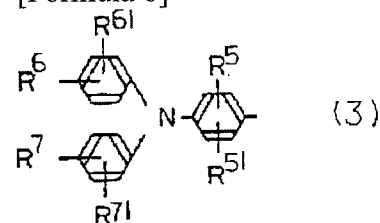
[0012] (;A the phenyl group which may differ and may have the alkyl group or the substituent is indicated to be, and n indicates the integer of 1-10 to be shows among a formula that R1 and R2 are the same or the radical expressed with the following type (2) or (3)) : [0013]

[Formula 5]



[0014] (Among a formula, R3, R4, R31, and R41 being the same or the phenyl group which may differ and may have the alkyl group or the substituent is shown, and l and m show the integer of 0-3) : [0015]

[Formula 6]



[0016] (the inside of a formula, and R5, R6, R7, R51, R61 and R71 are the same -- or it differs and a hydrogen atom, an alkyl group, an alkoxy group, an aryl group, or a cycloalkyl radical is shown) .

[0017]

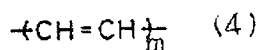
[Function] If light is irradiated by the photoconductive layer of an organic solar battery, while a charge is generated and an electron moves to a positive electrode, an electron hole will be poured into a charge transportation color, and will be moved to a negative electrode through this charge transportation color at the charge generating pigment in a photoconductive layer.

[0018] Here, the triphenyl benzene derivative (compound which is the radical as which A is expressed in the above-mentioned formula (2) in the above-mentioned general formula (1)) used for this invention has long electronic conjugated system, and its hole migration length of intramolecular is long. Therefore, compared with the charge transportation color currently indicated by JP,1-215070,A, it excels in hole migration capacity. Moreover, this triphenyl benzene derivative is excellent also in the stability over light. Therefore, by making a photoconductive layer contain by making this triphenyl benzene derivative into a charge transportation color, it excels in photoelectric conversion efficiency, and an organic solar battery with the stable property at the time of continuous duty is obtained.

[0019] Moreover, the triphenylamine derivative (compound which is the radical as which A is expressed in the above-mentioned formula (3) in the above-mentioned general formula (1)) used for this invention forms the phenyl group which the double bond of the radical expressed with the following formula (4) combines with this, and long conjugated double bond.

[0020]

[Formula 7]



[0021] For this reason, the above-mentioned triphenylamine derivative is excellent in hole migration capacity compared with the charge transportation color currently indicated by JP,1-215070,A. Therefore, the organic solar battery excellent in photoelectric conversion efficiency can be obtained by making a photoconductive layer contain by making this triphenylamine derivative into a charge transportation color. Moreover, this triphenylamine derivative is excellent also in compatibility with binder resin. Therefore, by making a photoconductive layer contain this, a uniform photoconductive layer can be formed and an organic solar battery with the stable property at the time of continuous duty can be obtained.

[0022] Especially the compound that has an alkyl group as a substituent among the above triphenylamine derivatives is excellent in compatibility with resin. Moreover, as for n, in the above-mentioned general formula (1), it is desirable that it is the integer of 1-3.

[0023]

[Best Mode of Carrying Out the Invention] The organic solar battery concerning this invention can be considered as a configuration as shown in drawing 1.

[0024] This organic solar battery A has a substrate 1, and the electrode 2, the photoconductive layer 3 and the transperence counterelectrode 4 by which the laminating was carried out one by one on this substrate 1.

[0025] In this structure, although light is irradiated from a transparent electrode 4 side, when a substrate 1 and an electrode 2 are translucency, light may be irradiated from a substrate 1 side. In that case, a transparent electrode 4 may be non-translucency. Moreover, in this structure, an under-coating layer may be prepared on a substrate 1 for the improvement in adhesion with an electrode 2 and a substrate 1.

[0026] As an ingredient of the above-mentioned substrate 1, metals, such as aluminum and stainless steel, paper, plastics, etc. can be used.

[0027] All can be used if it is the ingredient which forms the Schottky barrier with a photoconductive layer 3 as an ingredient of the above-mentioned electrode 2 and a transparent electrode 4. For example, the thing which dissolved conductive polymer; quaternary

ammonium salt, such as metal; polyacetylenes, such as aluminum, copper, and stainless steel, and polypyrrole, into the macromolecule; oxides, such as SnO₂ and ITO, etc. can be used. However, when translucency is needed, it is necessary to use a metaled translucent thin film, a transparent conductive oxide, etc.

[0028] The above-mentioned electrodes 2 and 4 are formed of vacuum deposition or sputtering.

[0029] Generally the above-mentioned photoconductive layer 3 contains binder resin, a charge generating pigment, and a charge transportation color.

[0030] As an organic charge transportation color contained in the above-mentioned photoconductive layer 3, the compound expressed with the above-mentioned general formula (1) is used.

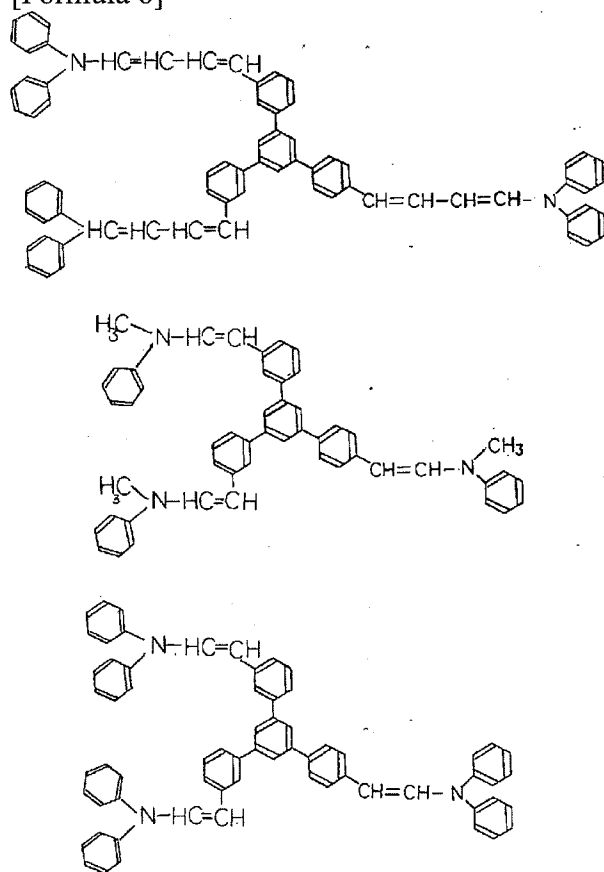
[0031] As the above-mentioned alkyl group, the straight chain or the branched alkyl groups of carbon numbers 1-6, such as a methyl group, an ethyl group, a propyl group, an isopropyl group, butyl, an isobutyl radical, t-butyl, a pentyl radical, and a hexyl group, are raised, for example.

[0032] Moreover, the low-grade alkyl group of the carbon numbers 1-6 above as a radical which may be permuted by the above-mentioned phenyl group; aryl groups, such as a straight chain of the carbon numbers 1-6, such as a methoxy group, an ethoxy radical, an isopropoxy group, a butoxy radical, a t-butoxy radical, and a hexyloxy radical, or a branched alkoxy group; phenyl group, a naphthyl group, an anthryl radical, and a phenan tolyl group, etc. are raised.

[0033] In the above-mentioned general formula (1), the following are raised, for example as a triphenyl benzene derivative which is the radical as which A is expressed in the above-mentioned formula (2).

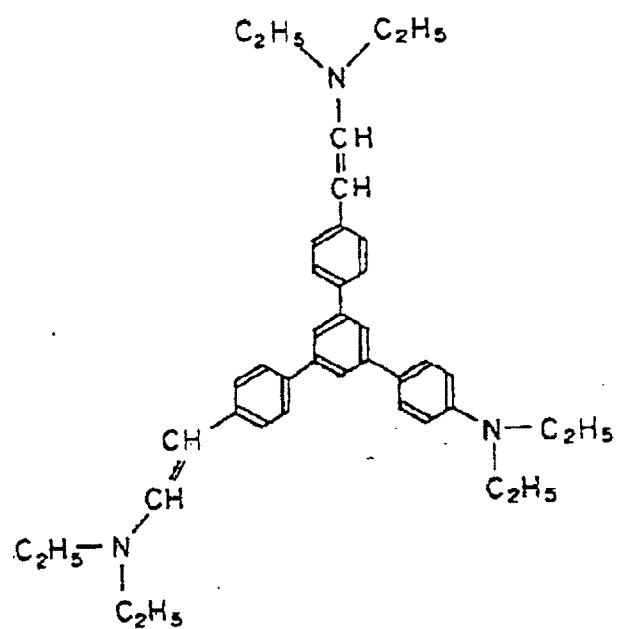
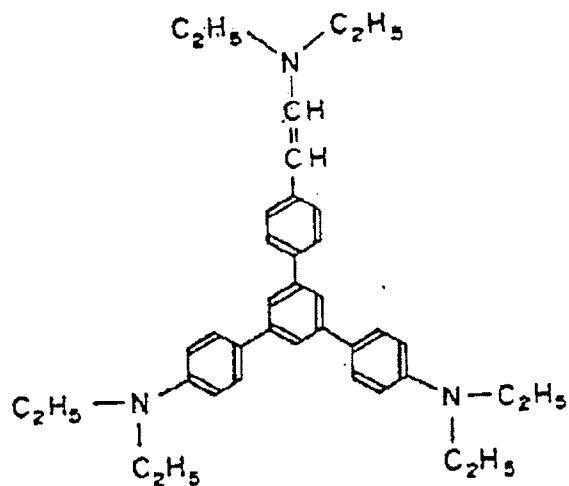
[0034]

[Formula 8]



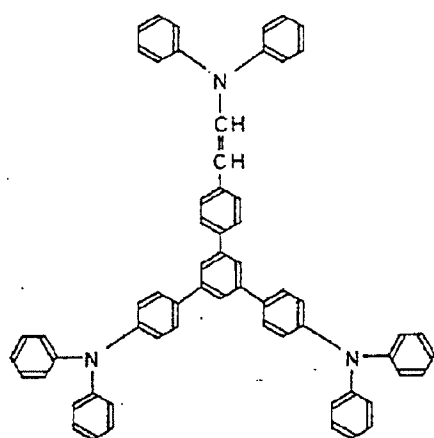
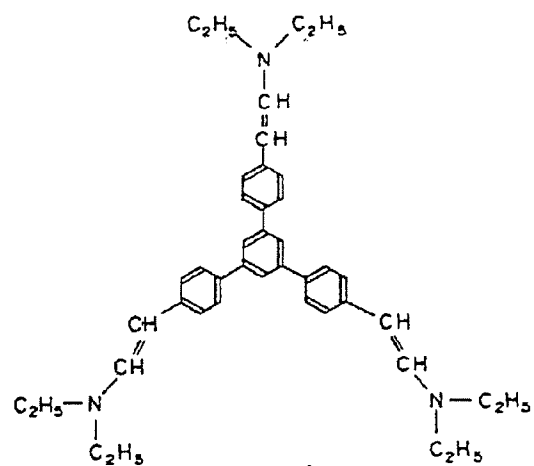
[0035]

[Formula 9]



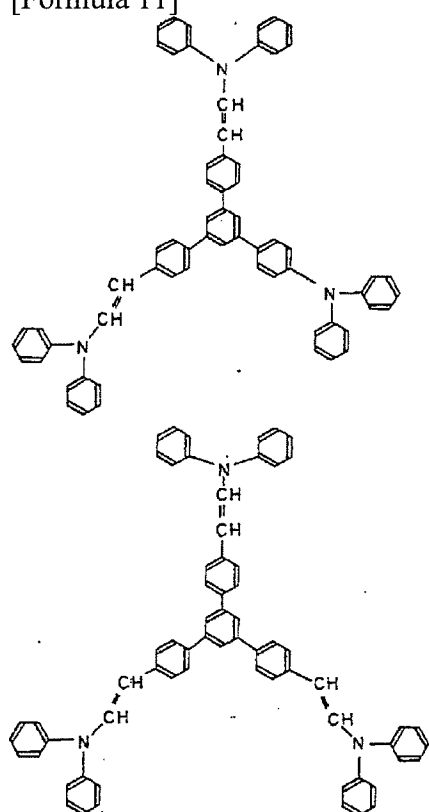
[0036]

[Formula 10]



[0037]

[Formula 11]

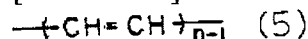


[0038] The above-mentioned triphenyl benzene derivative can be manufactured using various reaction processes. For example;

(1) Beforehand, a $-\text{CH}_2\text{CHO}$ radical is introduced through the radical expressed with the following type (5), and approach: to which this is made to react with a predetermined amine system compound is mentioned to the each 4th place of three phenyl groups of 1 of the main benzene ring, and the approach:(2) triphenyl benzene which introduces into the 3 or 5th place the phenyl group which has a predetermined permutation machine, respectively.

[0039]

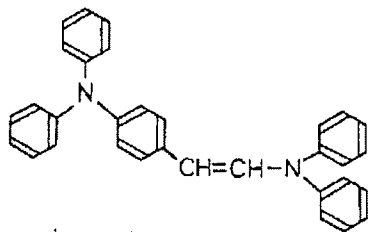
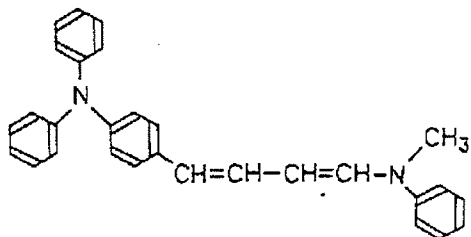
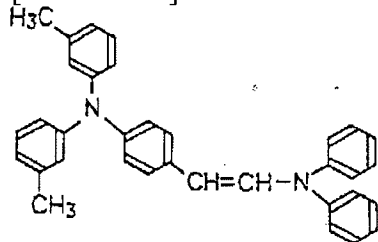
[Formula 12]



[0040] In the above-mentioned general formula (1), the following are mentioned, for example as a triphenylamine derivative which is the radical as which A is expressed in the above-mentioned formula (3).

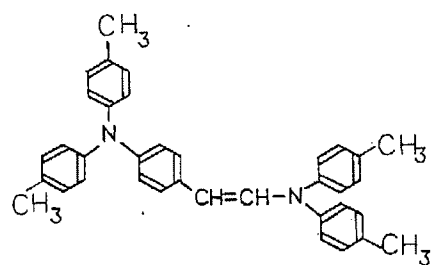
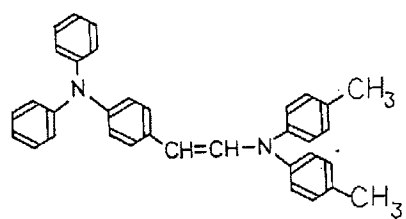
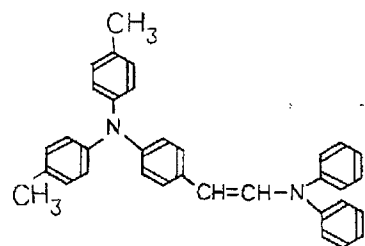
[0041]

[Formula 13]



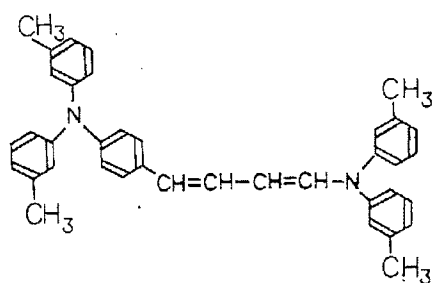
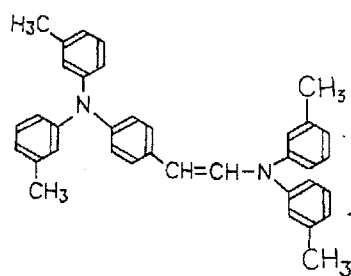
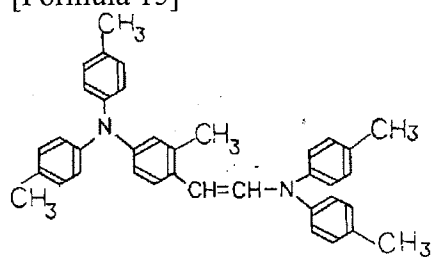
[0042]

[Formula 14]



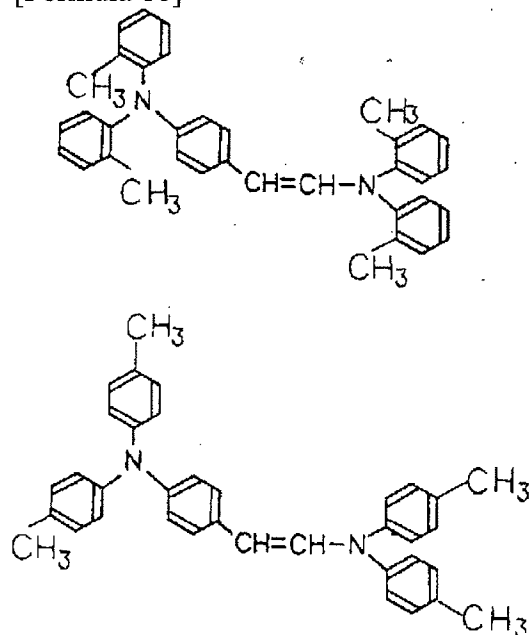
[0043]

[Formula 15]



[0044]

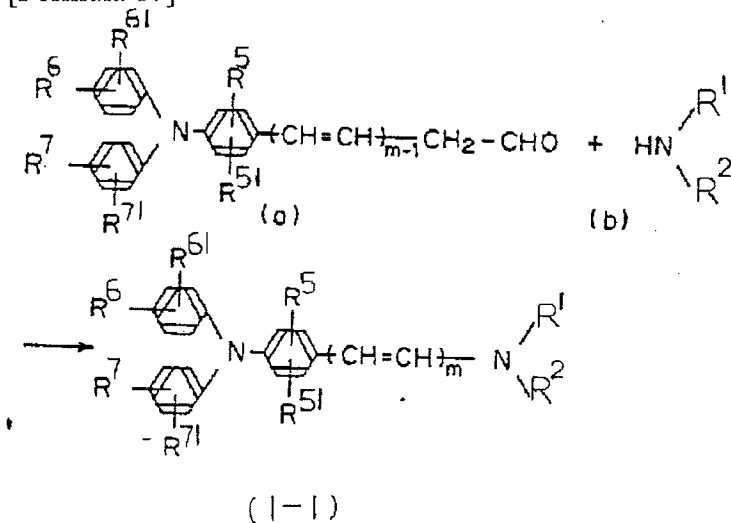
[Formula 16]



[0045] The above-mentioned triphenylamine derivative can be manufactured for example, in the following reaction formula (new experimental science lecture (publishing office: Maruzen Co., Ltd.) 14- refer to III and the 1417-1427th page).

[0046]

[Formula 17]



[0047] (R_1 , R_2 , R_5 , R_6 , R_7 , R_{51} , R_{61} , R_{71} , and n are similarly defined as the above among a formula).

[0048] That is, add an acetic acid, p-toluenesulfonic acid, etc., the amine system compound expressed with a formula (b) to the aldehyde system compound expressed with a formula (a) is made to react, and the above-mentioned triphenylamine system compound expressed with a formula (1-1) is obtained. This reaction can be performed by flowing back in a suitable solvent. As the above-mentioned solvent, benzene, toluene, a xylene, etc. are mentioned, for example.

[0049] Moreover, the compound expressed with the above-mentioned general formula (1) may be used independently, and can also be conventionally used combining other well-known charge transportation ingredients. As a well-known charge transportation ingredient, various

electronic suction nature compounds and an electron-donative compound can be used conventionally.

[0050] as the above-mentioned electronic suction nature compound -- diphenoquinone derivative [, such as 2 and 6-dimethyl-2' and 6'-JI tert-dibutyl diphenoquinone], MARONO nitril, thiopyran system compound, tetracyanoethylene, 2 and 4, 8-trinitro thioxan ton, 3, 4 and 5, and 7-tetra-nitroglycerine-9-full -- me -- non, a dinitrobenzene, a dinitro anthracene, a dinitro acridine, nitro anthraquinone, dinitro anthraquinone, a succinic anhydride, a maleic anhydride, a dibromo maleic anhydride, etc. are illustrated.

[0051] As an electron-donative compound, moreover, 2, 5-JI (4-methylamino phenyl), Styryl system compounds, such as oxadiazole system compounds, such as 1, 3, and 4-oxadiazole, and 9-(4-diethylaminostyryl) anthracene, Pyrazoline system compounds, such as carbazole system compounds, such as a polyvinyl carbazole, and 1-phenyl-3-(p-dimethylaminophenyl) pyrazoline, A hydrazone compound, a triphenylamine system compound, the Indore system compound, Nitrogen ring type compounds, such as an oxazole system compound, an isoxazole system compound, a thiazole system compound, a thiadiazole system compound, an imidazole system compound, a pyrazole system compound, and a triazole compound, and a condensed multi-ring type compound are illustrated.

[0052] these charge transportation ingredients -- one sort -- or two or more sorts are mixed and it is used. In addition, to use the charge transportation ingredient which has membrane formation nature, such as a polyvinyl carbazole, binder resin is not necessarily required.

[0053] As a charge generating pigment contained in the above-mentioned photoconductive layer 3, what has the good absorptivity of sunlight or the light is desirable, and what functions as an electron acceptor to the above-mentioned charge transportation color is desirable. For example, a selenium and selenium-tellurium, a selenium-arsenic, an amorphous silicon, Pyrylium salt, a thio pyrylium system pigment, a phthalocyanine system compound, an anthanthrone system compound, An anthanthrone pigment, a perylene system compound, a JIBENZU pyrene quinone pigment, A PIRATORON pigment, a tris azo pigment, a bis-azo pigment, an azo compound, a JISUAZO system compound, An indigo system compound, a thioindigo system compound, a triphenylmethane color system compound, The Indanthrene system compound, a toluidine system compound, a pyrazoline system compound, a KINAKU drine compounds system compound, a pyrrolo pyrrole system compound, unsymmetrical kino cyanine, kino cyanine, polycyclic condensed system coloring matter, porphyrin coloring matter, etc. can be used.

[0054] One sort or two sorts or more may be mixed and used for these charge generating pigments.

[0055] The above-mentioned charge generating pigment can be made into the particle of suitable crystal form by heating churning of milling dispersion liquid etc.

[0056] As binder resin which constitutes the above-mentioned photoconductive layer 3 For example, a styrene system polymer, a styrene-butadiene copolymer, a styrene acrylonitrile copolymer, A styrene-maleic-acid copolymer, an acrylic copolymer, a styrene-acrylic-acid copolymer, Polyethylene, an ethylene-vinylacetate copolymer, chlorinated polyethylene, A polyvinyl chloride, polypropylene, a vinyl chloride vinyl acetate copolymer, Polyester, a polyamide, alkyd resin, polycarbonates, polyarylate, Polysulfone, diallyl phthalate resin, ketone resin, polyvinyl butyral resin, Photo-setting resins, such as epoxy acrylate and urethane-acrylate, etc. are mentioned to thermoplastics, such as polyether resin and polyurethane resin, silicone resin and an epoxy resin, phenol resin, a urea-resin, melamine resin, the other thermosetting resin of cross-linking, and a pan. One sort or two sorts or more can be mixed and used for these binder resin.

[0057] The organic solar battery of this invention is the following, and can be made and produced.

[0058] First, coating liquid is prepared for the above-mentioned charge generating pigment, a

charge transportation color, binder resin, and a solvent using the approach of a homogenizer, a supersonic wave, a ball mill, a sand mill, attritor, a roll mill, or a paint shaker. In this coating liquid, the above-mentioned charge generating pigment is distributed in the shape of a particle, and the above-mentioned charge transportation color is dissolved in homogeneity.

[0059] As the above-mentioned solvent, various organic solvents are usable. For example, alcohols, such as a methanol, ethanol, isopropanol, and a butanol, Aliphatic series system hydrocarbons, such as n-hexane, an octane, and a cyclohexane, benzene, Aromatic hydrocarbon, such as toluene and a xylene, dichloromethane, a dichloroethane, Halogenated hydrocarbon, such as a carbon tetrachloride and a chlorobenzene, wood ether, Diethylether, a tetrahydrofuran, ethylene glycol wood ether, Ester, such as ketones, such as ether, such as diethylene-glycol wood ether, an acetone, a methyl ethyl ketone, and a cyclohexanone, ethyl acetate, and methyl acetate, dimethyl formaldehyde, dimethylformamide, dimethyl sulfoxide, etc. are raised. These solvents can mix and use one sort or two sorts or more.

[0060] Next, the laminating of the above-mentioned coating liquid is carried out on an electrode 2 by casting, dipping, etc., and a photoconductive layer 3 is formed.

[0061] In addition, the above-mentioned photoconductive layer 3 can be made to contain various additives for a generation of carriers, impregnation, and the improvement in transportability. For example, diphenyl, chlorination diphenyl, terphenyl, halo naphthoquinones, dibutyl phthalate, dimethyl glycol phthalate, dioctyl phthalate, triphenyl phosphoric acid, a methylnaphthalene, acetyl naphthalene, a benzophenone, chlorinated paraffin, dilauryl thiopropionate, 3, 5-dinitro salicylic acid, and various fluorocarbon are mentioned.

[0062] Furthermore, in order to improve the dispersibility of a charge transportation color or a charge generating pigment, dyer nature, etc., a surface active agent, a leveling agent, etc. may be used.

[0063] Here, as for 2 - 20 weight section and especially the phenylenediamine system compound expressed with 3 - 15 weight section and the above-mentioned general formula (1), it is [a charge generating pigment] especially desirable to the above-mentioned binder resin 100 weight section the 40 - 200 weight section and that it is the 50 - 150 weight section.

Moreover, as for especially the thickness of a photoconductive layer, it is desirable that it is about 0.5 micrometers 0.1-2 micrometers.

[0064]

[Example] Hereafter, an example and the example of a comparison are given and this invention is explained to a detail.

[0065] (Examples 1-11 and examples 1-5 of a comparison) On a 100mmx100mm glass plate, vacuum deposition of the aluminum electrode (70% of translucent rates) with a thickness of 500nm is carried out.

[0066] Next, as a charge generating pigment and a charge transportation color, every [10 weight sections], the polyvinyl-butylal-resin (S lek BM-1, Sekisui Chemical Co., Ltd. make) 10 weight section, and the cyclohexanone 50 weight section were mixed, and mixed distribution of the compound shown in Table 1 was carried out with the ball mill using a glass bead with a diameter of 1mm for 24 hours. Using the spin coat method, the obtained dispersion liquid were applied on the above-mentioned aluminum electrode, were dried for 30 minutes at 100 degrees C, and the photoconductive layer with a thickness of 0.5 micrometers was formed.

[0067] Moreover, the 20mmx20mm golden electrode was vapor-deposited in thickness of 2000A.

[0068] The organic solar battery was obtained according to the above-mentioned process.

[0069] (Evaluation trial) The open-circuit electrical-potential-difference VOC[of the organic solar battery obtained in each examples 1-11 and the examples 1-5 of a comparison] (V) short circuit current ISC (mA) and the maximum photoelectric conversion efficiency EMAX

at the time of 1kohm addition (%) were measured on condition that the following.
 [0070] light source: -- tungsten lamp quantity of light: -- a 200 lux test result is collectively shown in Table 1. Moreover, the chemical structure type of the charge generating pigment shown all over Table 1 and a charge transportation color is shown below.

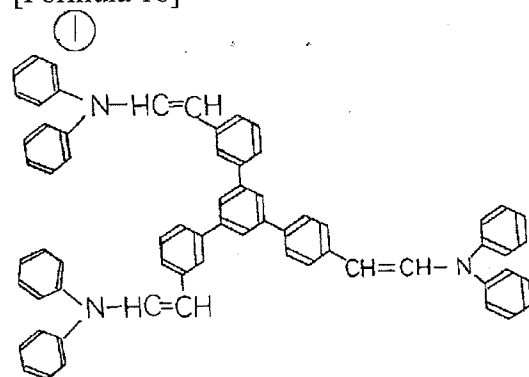
[0071]

[Table 1]

	電荷発生顔料	電荷輸送染料	V _{oc}	I _{sc}	E _{max}
実施例 1	X y P T C	①	1.20 V	1.15 mA	0.58 %
実施例 2	M e P T C	①	1.15 V	1.10 mA	0.56 %
実施例 3	x-H ₂ P c	①	1.10 V	1.10 mA	0.55 %
実施例 4	T i O - P c	①	1.20 V	1.20 mA	0.61 %
実施例 5	X y P T C	②	1.25 V	1.15 mA	0.57 %
実施例 6	X y P T C	③	1.15 V	1.10 mA	0.56 %
実施例 7	X y P T C	④	1.15 V	1.05 mA	0.53 %
実施例 8	M e P T C	④	1.10 V	1.00 mA	0.50 %
実施例 9	x-H ₂ P c	④	1.00 V	0.95 mA	0.48 %
実施例 10	T i O - P c	④	0.95 V	0.95 mA	0.58 %
実施例 11	X y P T C	⑤	1.15 V	1.10 mA	0.56 %
比較例 1	X y P T C	D E H	0.68 V	0.33 mA	0.17 %
比較例 2	M e P T C	D E H	0.62 V	0.28 mA	0.14 %
比較例 3	x-H ₂ P c	D E H	0.59 V	0.26 mA	0.13 %
比較例 4	T i O - P c	D E H	0.74 V	0.36 mA	0.19 %
比較例 5	X y P T C	D P D E B	0.70 V	0.35 mA	0.18 %

[0072]

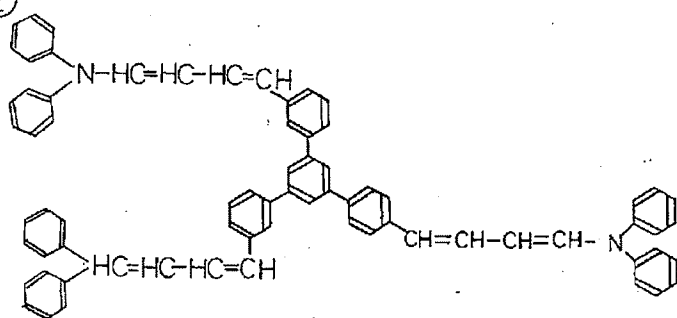
[Formula 18]



[0073]

[Formula 19]

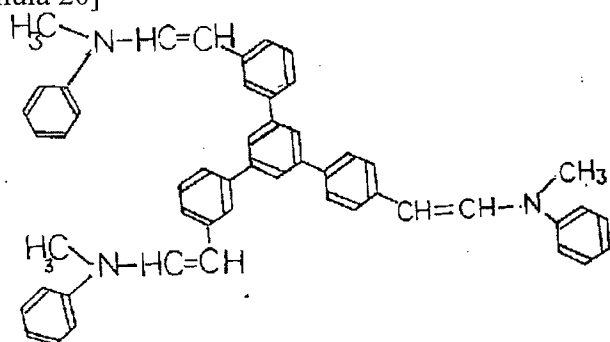
②



[0074]

[Formula 20]

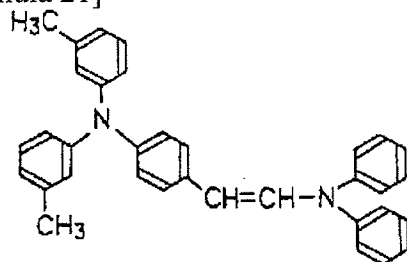
③



[0075]

[Formula 21]

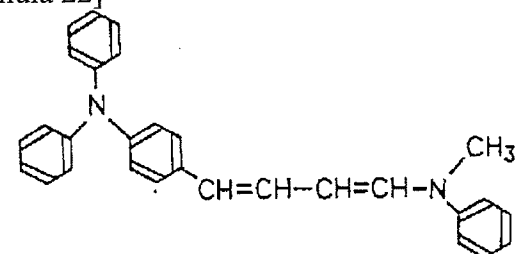
④



[0076]

[Formula 22]

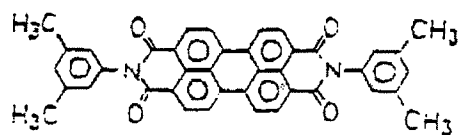
⑤



[0077]

[Formula 23]

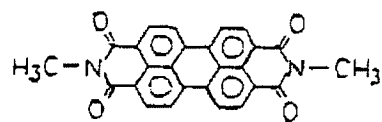
XyPTC



[0078]

[Formula 24]

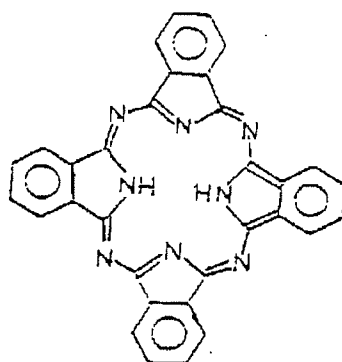
MePTC



[0079]

[Formula 25]

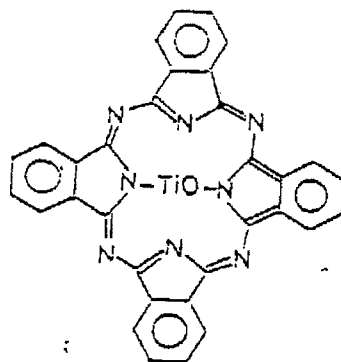
x-H₂Po



[0080]

[Formula 26]

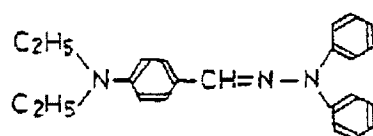
TiO-Po



[0081]

[Formula 27]

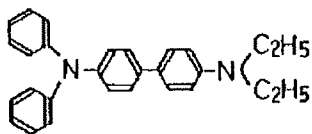
DEH



[0082]

[Formula 28]

DPDEB



[0083] From these test results, each organic solar battery of this invention shows the good value compared with the conventional organic solar battery in the open-circuit electrical-potential-difference VOC(V) short circuit current ISC (mA) and the maximum photoelectric conversion efficiency EMAX at the time of 1kohm addition (%).

[0084] Moreover, about the stability over light, it excels compared with the conventional organic solar battery, and a stable property is acquired at the time of continuous duty.

[0085]

[Effect of the Invention] According to this invention, compared with the conventional organic solar battery, it excels in photoelectric conversion efficiency and the organic solar battery which has a stable property at the time of continuous duty can be obtained so that clearly from the above explanation.

[Translation done.]